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# Failure Investigation of 3M Series 900 Static Eliminators

Prepared by C. Czajkowski

Brookhaven National Laboratory

Prepared for
U.S. Nuclear Regulatory
Commission

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# Failure Investigation of 3M Series 900 Static Eliminators

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Prepared by
C. Czajkowski

Brookhaven National Laboratory
Upton, NY 11973

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**Division of Industrial and Medical Nuclear Safety**
**Office of Nuclear Material Safety and Safeguards**
**U.S. Nuclear Regulatory Commission**
**Washington, DC 20555**
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**Region I**
**U.S. Nuclear Regulatory Commission**
**475 Allendale Road**
**King of Prussia, PA 19406**

## ABSTRACT

Numerous instances of facility contamination by polonium-210 microspheres have been reported. These contamination events appear to be caused by leakage or ejection of the radioactive material from static elimination devices manufactured by the Minnesota Mining and Manufacturing Company (3M). The Ashland Chemical Company (Easton, Pennsylvania) was the first facility determined to be contaminated and was subsequently subjected to a comprehensive review by the U. S. Nuclear Regulatory Commission (USNRC). Therefore, devices from this facility were chosen for examination.

A failure investigation has been performed on six static eliminators. The investigation consisted of visual inspection, sectioning and scanning electron microscopy of six devices. It is concluded that non-uniform and imperfect microspheres appear to have been manufactured and installed in devices. Rough handling may induce loosening of the microspheres. The epoxy binder used in device manufacture may not be suitable for the varied service conditions.


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## 1. INTRODUCTION

On January 25, 1988, the U. S. Nuclear Regulatory Commission (USNRC) issued an order to the Minnesota Mining and Manufacturing Company (3M) suspending the distribution of certain static eliminating devices containing polonium-210. This suspension of distribution was prompted in part by the fact that Ashland Chemical Company reported (to the NRC) radioactive polonium-210 contamination at its facilities in Easton, Pennsylvania and Dallas, Texas, stemming from leakage of these devices. Devices affected by the NRC order are Model Numbers 902, 902F, 906, and 908. The Model 908 devices were those used at the Ashland Chemical facilities.

Static eliminators of this type contain radioactive polonium-210 which is fused into small (30-35 μm diameter) spheres, sometimes referred to as microspheres. These microspheres are composed of a zirconium pyrophosphate ($ZrP_2O_7$) ceramic. The polonium-210 is purified [1] and adsorbed on the outer layers of the microspheres which are then fired at 700°C to form a ceramic bead containing polonium-210. After firing, the microspheres are plated with a nickel coating of approximately 1 μm. These microspheres are then incorporated into an epoxy binder. The epoxy is then installed into the metal tube used as the static eliminator. The release of alpha particles ionizes compressed air blown through the device which reduces the static charge on surfaces onto which the ionized air is directed (Figure 1).

In order to independently ascertain potential failure modes of the 900 series 3M static eliminators, the USNRC contracted with Brookhaven National Laboratory (BNL) for a failure investigation of a sample of the eliminators used at the Ashland Chemical, Easton, Pennsylvania facility. This failure investigation was to encompass the following.

1. Receipt and visual inspection of new and failed devices;
2. Wipe surveys of new and failed devices to check radioactivity levels;
3. Selection of two new and at least one failed device from each use environment (6 total).

For each of these six devices, the following examination protocol was adhered to:

a. Conduct visual examination/optical photography
b. Disassemble/section devices
c. Optical microscopic examination of device internals/photography
d. Scanning electron microscopic (SEM) examination of device internals.

This report is the documentation of the failure investigation.



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## 2. RECEIPT/VISUAL INSPECTION/WIPE SURVEY/TAP TEST

A total of thirty-four type 908 series 3M static eliminators were sent to BNL from the Ashland Chemical Company. A thirty-fifth (as yet unboxed) specimen was sent to BNL after the investigation was complete. Twelve of the static eliminators received were new, unused devices (samples 1-12, Table 1). Two of these new devices D74465 and D74471 were chosen at random for sectioning. The remainder of the devices (other than 12 new ones) had seen service at the Easton, Pennsylvania facility. Samples "B," "I," D64601, and D74410 were also chosen by the BNL investigator to be reasonably representative of the Ashland Chemical's service environment (Table 1: Nos. 14, 21, 29, and 30).

Samples "B" and "I" (ID #14 and 21 in Table 1) were used in Ashland's automatic bottle washing facility. The service conditions were compressed air blown through the devices at 90 psi in a moist air environment at 140°F (60°C).

Samples D64601 and D74410 were used at Ashland's manual filling station and had been exposed to dry compressed air blown through the devices (5-65 psi) and ambient temperature and moisture conditions.

The six specimens chosen as the BNL samples were photographed (Figures 2-7). Five of the devices appeared to be jacketed in stainless steel while D64601 (Figure 4) gave the appearance of being made from a copper alloy (probably brass).

Three of the devices appeared to have scratches on them from service (Figures 4, 6, and 7) with device "B" having gouges on the outside surface similar to those which pliers might produce (Figure 6).

Each of the devices was dry wiped (smeared) on the exterior surface. The wipes were counted in an alpha scintillator. The results of this survey are shown in Table 1. Eleven of the thirty-four devices were contaminated including six of the twelve new devices. The highest recorded contamination level was attributed to specimen D74469 - a new, unused device that was still sealed in the original 3M plastic bag.

Since it was apparent that normal handling could possibly loosen the polonium microspheres from the devices; D74465 and D74471 were subjected to a "TAP TEST." The test consisted of taping over both openings of the static eliminator (male and female threaded ends), then giving a single sharp tap on each end of the device against a hard surface. The intent was not to damage the device but only to ascertain if rough handling could loosen the microspheres from the epoxy matrix.

The resultant four pieces of tape were then counted in the alpha scintillator. Specimen D74465 displayed no measurable activity on either tape sample while D74471 had an activity of 339,100 cpm (916,486 dpm) on the female end tape specimen (the male end showing no activity). This activity (916,486 dpm) equates to approximately 0.4 microcuries deposited on the tape. This appeared to be a clear indication that rough handling of the devices might be contributing to the contamination problem.

Table 1 Results of Wipe (Dry) Tests Air Ionizers Received at BNL

| Sample | Identification | Gross cpm | Net cpm | dpm |
|---|---|---|---|---|
| 1. D74464 | (loose) | 0 | - | - |
| 2. D74465 | (packaged) | 0 | - | - |
| 3. D74466 | (packaged) | 1 | 1.0 | 2.7 |
| 4. D74467 | (loose) | 0 | - | - |
| 5. D74468 | (packaged) | 0 | - | - |
| 6. D74469 | (packaged) | 142 | 142 | 383.8 |
| 7. D74470 | (loose) | 1 | .8 | 2.2 |
| 8. D74471 | (packaged) | 0 | - | - |
| 9. D74472 | (packaged) | 2 | 2.0 | 5.4 |
| 10. D74473 | (loose) | 4 | 3.8 | 10.3 |
| 11. D74474 | (loose) | 2 | 1.8 | 4.9 |
| 12. D74475 | (loose) | 0 | - | - |
| 13. A | | 0 | - | - |
| 14. B | | 0 | - | - |
| 15. C | | 1 | 1.0 | 2.7 |
| 16. D | | 0 | - | - |
| 17. E | | 0 | - | - |
| 18. F | | 0 | - | - |
| 19. G | | 0 | - | - |
| 20. H | | 0 | - | - |
| 21. I | | 0 | - | - |
| 22. J | | 0 | - | - |
| 23. K | | 0 | - | - |
| 24. L | | 0 | - | - |
| 25. M | | 0 | - | - |
| 26. N | | 0 | - | - |
| 27. D62253 | | 0 | - | - |
| 28. D62251 | | 7 | 6.8 | 18.4 |
| 29. D64601 | | 0 | - | - |
| 30. D74410 | | Not recorded | | 72 |
| 31. Bag 4 - Label Torn | | 11 | 10.8 | 29.2 |
| 32. Bag 4 - Unmarked | | 0 | - | - |
| 33. Bag 4 - Unmarked | | 0 | - | - |
| 34. Box 3 - Unmarked | | Not received | | |
| 35. D62256 (air gun) | | 13 | 12.8 | 34.6 |

## 3. EVALUATION OF EPOXY AND NON-RADIOACTIVE MICROSPHERES

Samples of two types of epoxy (black and amber) and a vial of non-radioactive microspheres (fused and nickel coated but no polonium-210) were received at BNL and examined by scanning electron microscopy (SEM).

The epoxy blanks were sputter coated prior to examination by the SEM.

Note:

> Insulating materials or nonconducting particles build up a space charge region by accumulation of absorbed electrons which deflect the incident beam of the scanning electron microscope. This deflection produces intense image distortion. These charging effects can be minimized by applying conductive coatings to the specimens. This coating ideally should be thick enough to provide a conductive path, yet thin enough not to mask any fine details of the specimen. Various materials can be applied, e.g., carbon, gold, palladium, platinum, silver, aluminum, or copper by either high vacuum evaporation or cathode sputtering. For the purposes of this investigation, a gold/palladium alloy was used to cathode sputter the samples to a thickness of approximately 300A.

Figures 8 through 11 are SEM photographs of the two blanks examined (black and amber epoxy). Both of the blanks appeared to be continuous in nature with no cracks visible. Discussions with USNRC personnel disclosed that the addition of 2% carbon black was the only difference between the compositions of the two epoxies.

A small sample of some of the non-radioactive microspheres was poured out onto a strip of masking tape, sputter coated and then examined by the SEM. Figures 12 through 15 are typical areas examined. It is apparent from the photographs that a significant number of the microspheres are in a cracked, split or otherwise damaged condition. Figures 13 and 15 are higher magnification fractographs of the types of cracking found on the microspheres.

An energy dispersive spectrographic (EDS) scan was performed on these microspheres which confirmed that they had indeed been nickel coated.

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## 4. SECTIONING/MACROPHOTOGRAPHY/SEM STUDIES

The six sample static eliminators were sectioned by hand cutting the devices lengthwise with a mini-hacksaw until the jacketing material was penetrated. A single edged straight razor blade was then used to cut the epoxy tape in order to open the various devices' internals for inspection.

The devices were then evaluated under a stereo microscope. During this study, it was determined that the depth of field on the curved surfaces of the devices was limited to the extent that meaningful optical photomicrographs could not be taken without removal and flattening of the epoxy tape. In lieu of flattening the tape, it was decided to perform macrophotography of the devices' internals and rely on scanning electron microscopy of the specimens' features. After visual inspection and macrophotography, one half of each cut device was sputter coated to a thickness of 300Å and then evaluated with the SEM.

### Examination of Device D74465

The first static eliminator sectioned and evaluated was the new (manufactured 1/12/88) device identified as D74465. It is clear in Figure 16 that the black epoxy appeared to be intact with no obvious degradation of the epoxy binder or the polonium-210 microspheres (grey circle in black matrix). Optical microscopy revealed small portions of the polonium-210 circle to have discrete areas where no adhesion of the microspheres appeared to have occurred. Figure 17 is a low magnification SEM photograph of a typical area of the specimen. In the upper left side of the photograph there is an area with no microspheres evident. This appeared to be one of the areas where no adhesion had taken place. There were various cracked and broken microspheres observed on this specimen (Figures 18 and 19) and apparent areas of microsphere decohesion (Figure 20) were also seen.

### Examination of Device D74471

Figure 21 is a macrophotograph of the cut and opened static eliminator D74471. The device appeared to be intact, with no obvious visual defects or abrasions on either the epoxy or the area of polonium microsphere adhesion. Optical microscopy again revealed areas where the microspheres were absent from the epoxy.

SEM evaluation of the microspheres (after the "TAP TEST") confirmed that many areas of the epoxy circle were not uniformly covered with microspheres (Figure 22). Additionally, apparent areas of cracking (Figure 23) were revealed on the periphery of the circle of microspheres. Damaged microspheres (Figure 24) and microsphere decohesion were also noted on this device.

### Examination of Device D64601

The brass jacketed device D64601 had a similar appearance visually (epoxy and polonium-210 microspheres) to that observed on samples D74465 and D74471. There did not appear to be gross defects or damage visible to the naked eye (Figure 25). The SEM showed (Figure 26) that the epoxy binder was intact, with no evidence of cracking outside the circle of polonium microspheres.

Further examination in the area of the microspheres, however, revealed cracked and broken microspheres, decohesion of the microspheres and cracking of the epoxy matrix surrounding the microspheres (Figures 27 and 28). This particular device had been used at the manual filling station where 5-65 psi dry compressed air is blown through the device and ambient temperature and humidity conditions are present.

Examination of Device D74410

The second device used at the manual filling station (D74410) had an area within the circle of polonium microspheres (Figure 29) which appeared to have missing material. Although this area may have been the result of the cutting/sectioning operations, it is believed that the area is characteristic of original manufacture, since no additional marring or disruption of the epoxy/microsphere matrix was found.

Optical microscopy revealed hairline cracks on the epoxy binder and microspheres positioned outside the circle where polonium-210 microspheres are normally affixed. Both of these conditions are documented on Figures 30 and 31. Additionally, there appeared to be areas where microspheres may have dislodged from the epoxy (Figure 30).

Examination of Device "B"

Static eliminator "B" was obtained from the automatic bottle washing facility at Ashland Chemical. The device was subject to compressed air at 90 psi, at temperature 140°F and moist air.

After sectioning, the visual examination of the inside surfaces of the device disclosed a significant discoloration in the epoxy material (Figure 32). This discoloration extended to the very edges of the epoxy material. Instead of the normal blackened appearance of the epoxy, this device had a bleached (wheat colored) appearance.

Optical microscopy disclosed large areas of apparently missing microspheres and a "reptile skin" like appearance to the epoxy material. Additionally, damaged and cracked microspheres were also observed.

Scanning electron microscopy revealed many areas of missing microspheres from the circle (Figures 33 and 34) with the "reptile skin" cracking of the epoxy very much in evidence (Figure 35). Some of the microspheres showed significant damage, as seen in Figures 36 and 37.

Various areas where the microspheres were missing were examined by the SEM. Figure 38 is a typical area showing the location of an absent microsphere. The absence of swelling or blistering in this area suggests that radiation damage to the epoxy (although a possible contributor) is not the prime method of microsphere dislodgement.

Examination of Device "I"

A second static eliminator (Device "I") from the bottle washing facility was examined and had the same discolored appearance (Figure 39) as Device "B."

In this specimen, however, the epoxy cracking ("reptile-skin effect") was clearly visible to the naked eye (arrows).

SEM pictures of this device (Figures 40-44) showed significantly more damage to the epoxy binder, i.e., holes are quite evident (Figures 40, 41, 43). The epoxy cracking appeared to continue under the polonium microspheres as shown in Figure 42. On this device, the epoxy seemed to have suffered greater separation (possibly shrinkage) in the cracks (Figures 43 and 44). The decohesion of microspheres and damage to microspheres was also quite evident on this device.

## 5. DISCUSSION

The potential safety health hazards from industrial use of static eliminations (polonium containing) have been examined in New Zealand [2]. This paper described various immersion tests performed on both rigid and flexible (the active surface being curved) type static eliminators. Various solvents were used (including water) in the testing. After the required immersion time had elapsed, the source was removed from the solvent, which was acidified and filtered. In the case of the sources in water (after 14 days), the measured filter activity (primarily discrete microspheres) was 100 nCi which shows that the adhesive which secures the microspheres in place was definitely affected by the water. The observations of this investigation appear to echo a need for a more careful look at the environmental conditions under which the static eliminators may be operated.

A previously mentioned report [1] stated that the microspheres themselves were sample tested in solutions of both 3N HCl (hydrochloric acid) and 1% EDTA (ethylenediamine tetraacetic acid). However, the epoxy binder may not have suffered such rigorous testing.

The observations of cracked and damaged microspheres on non-radioactive samples and on new, unused devices/reinforces the hypothesis that quality control of microsphere manufacturing methods deserves closer scrutiny.

The deposition of 0.4 μCi on a tape sample after a "TAP TEST" suggests that rough handling of these devices may initiate a contamination incident.

The dry wipe tests performed on various new (still in the original plastic envelopes) devices is a clear indication that some of these devices may have been a source of contamination in the "as received" condition.

The observed cracks in the epoxy of the new device (D74471) point to the area of epoxy quality control. This coupled with the widespread cracking of the epoxy in three of the devices examined (1 at ambient conditions; 2 at 140°F and moist air) suggests that the epoxy binder may not be suitable for the current service conditions.

## 6. CONCLUSIONS

The previous discussion and report observations have led to the following conclusions:

1. The manufacture of the 3M microspheres do not consistently produce uniform and perfect spheres. There is evidence that cracked and broken microspheres are being produced and incorporated into static eliminating devices.

2. Rough handling of the devices may initiate polonium-210 leakage during or prior to service.

3. "As received" devices may be potential sources of contamination (exterior surfaces).

4. The epoxy binder appears to suffer significant environmental and material damage even under ambient conditions. The effect of solvents, heat, moisture or vibration may accelerate this damage. An evaluation of the "suitability for service" of this epoxy binder appears warranted. The breakdown of the epoxy binder appears to be a significant contributor to the contamination by loose microspheres or microsphere fragments.

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## 7. REFERENCES


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2. Robertson, M. K., and Randle, M. W., Health Physics, Vol. 26, pp. 245-250, March 1974.

## 8. ACKNOWLEDGEMENTS

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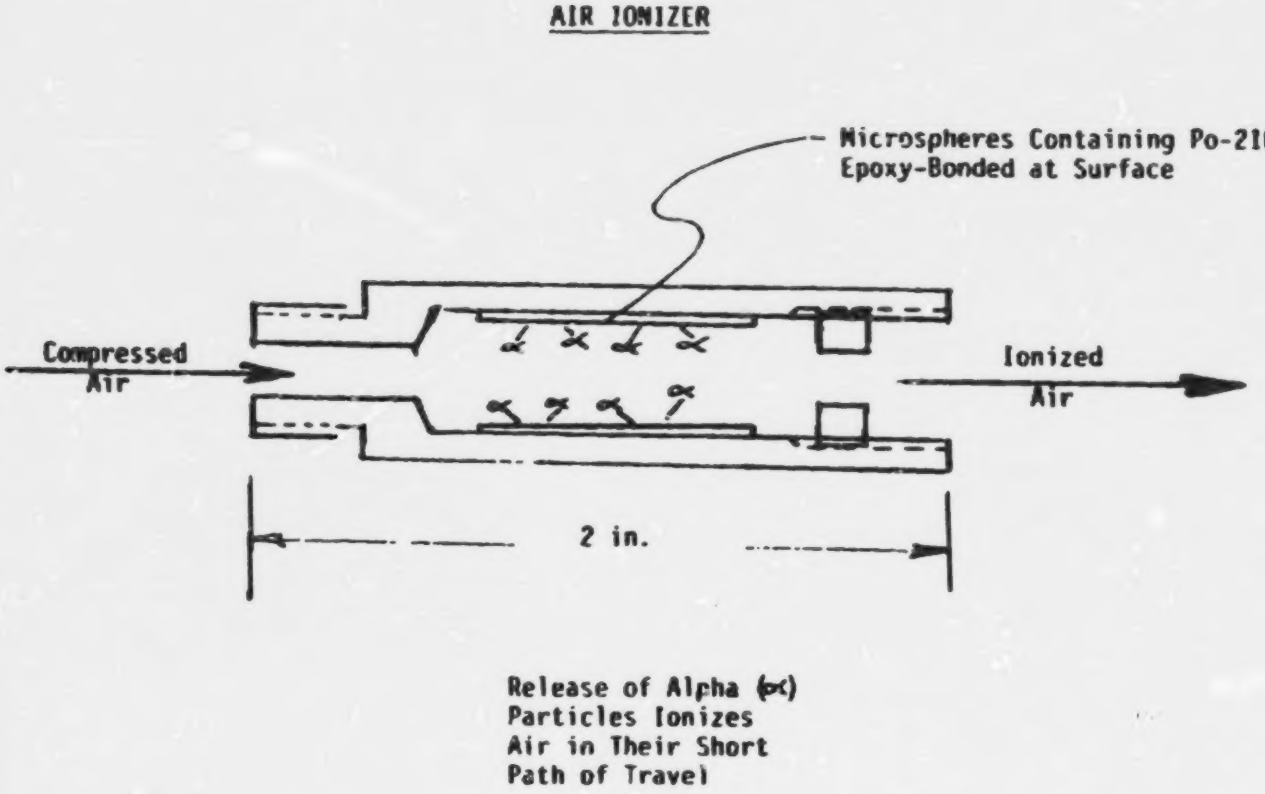


Figure 1 Sketch of a typical series 900 3M static eliminator.



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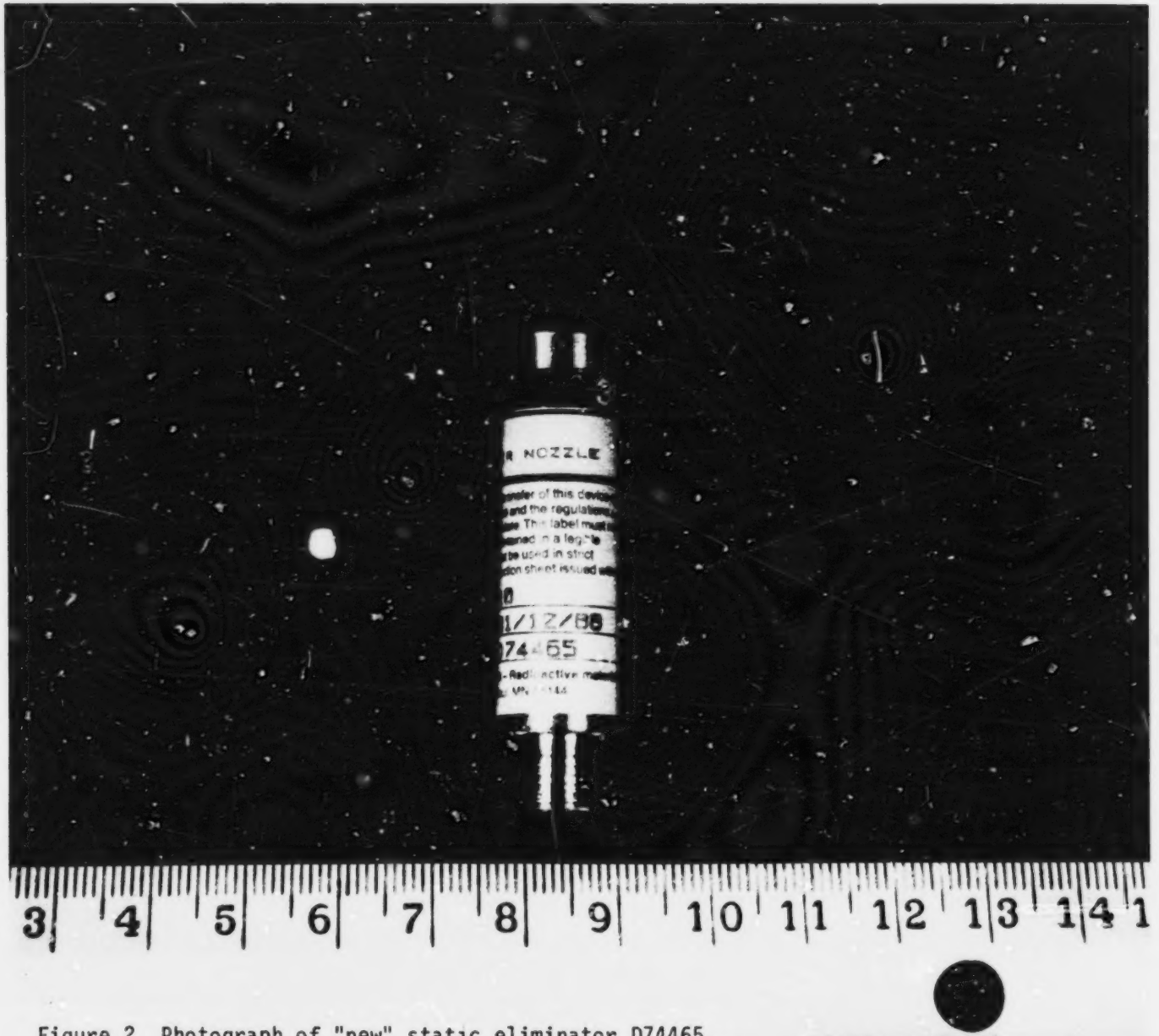


Figure 2 Photograph of "new" static eliminator D74465.

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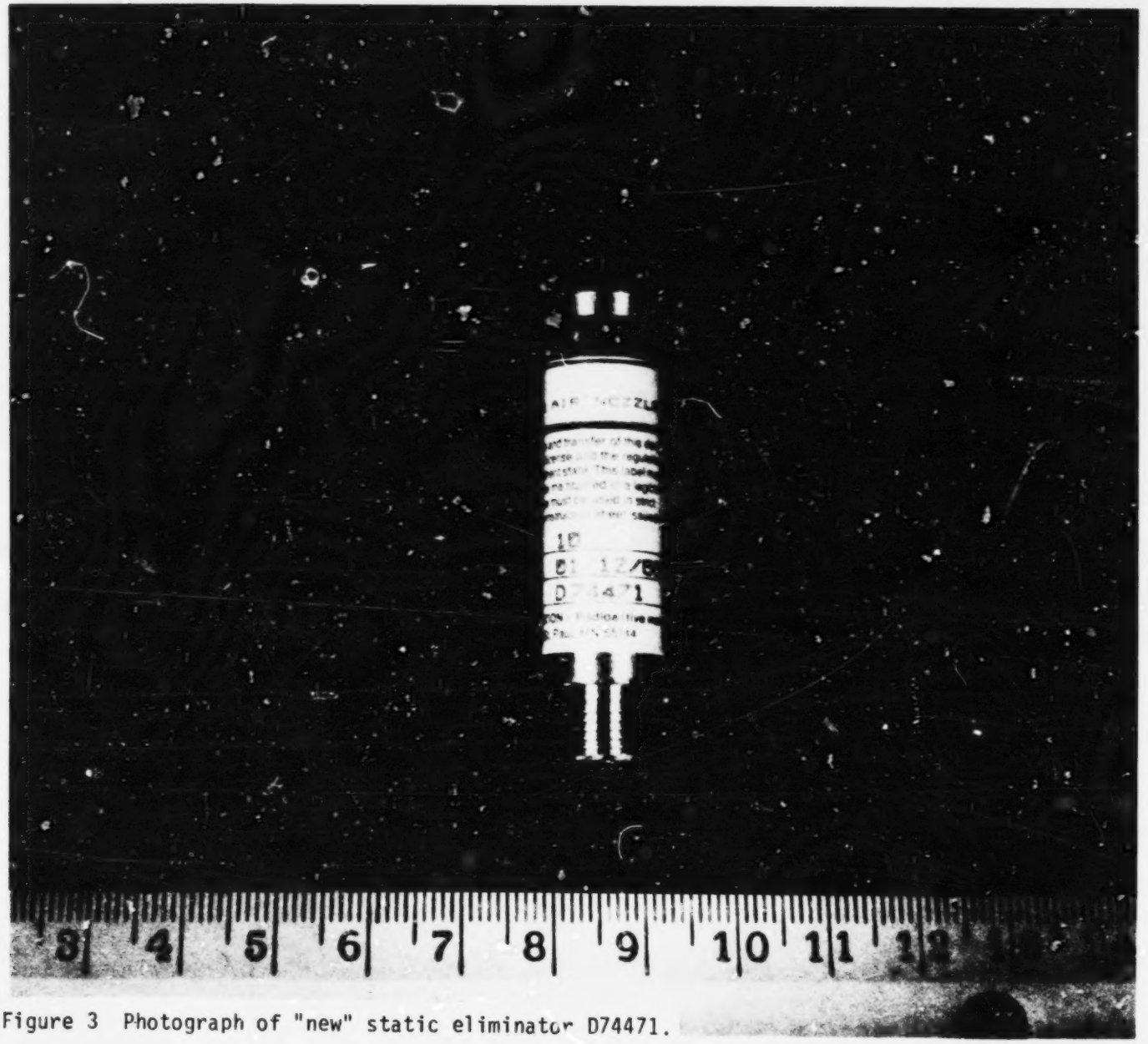


Figure 3 Photograph of "new" static eliminator D74471.

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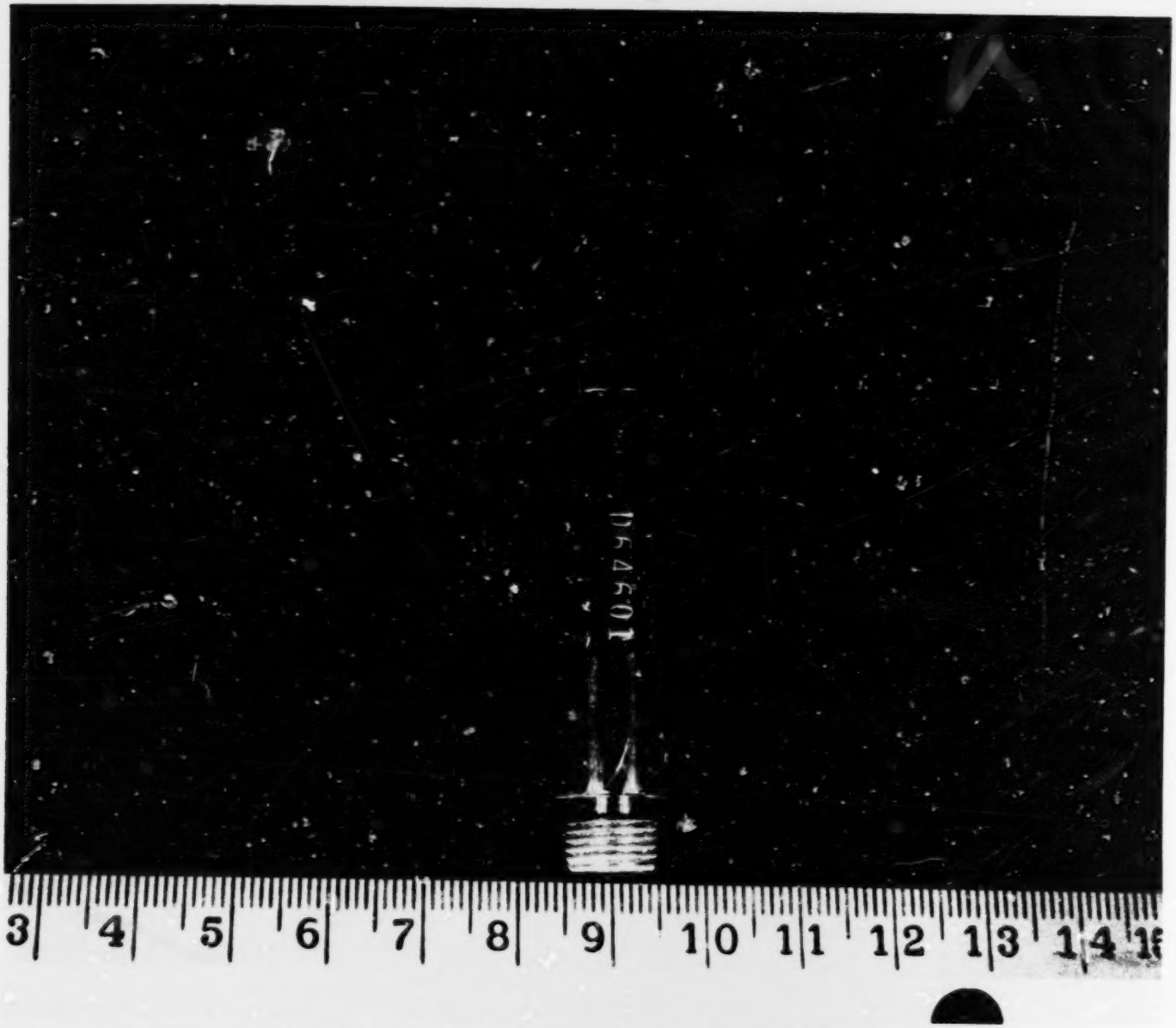


Figure 4 Photograph of static eliminator D64601 from the manual filling station. The case on this device appeared to be made from brass.

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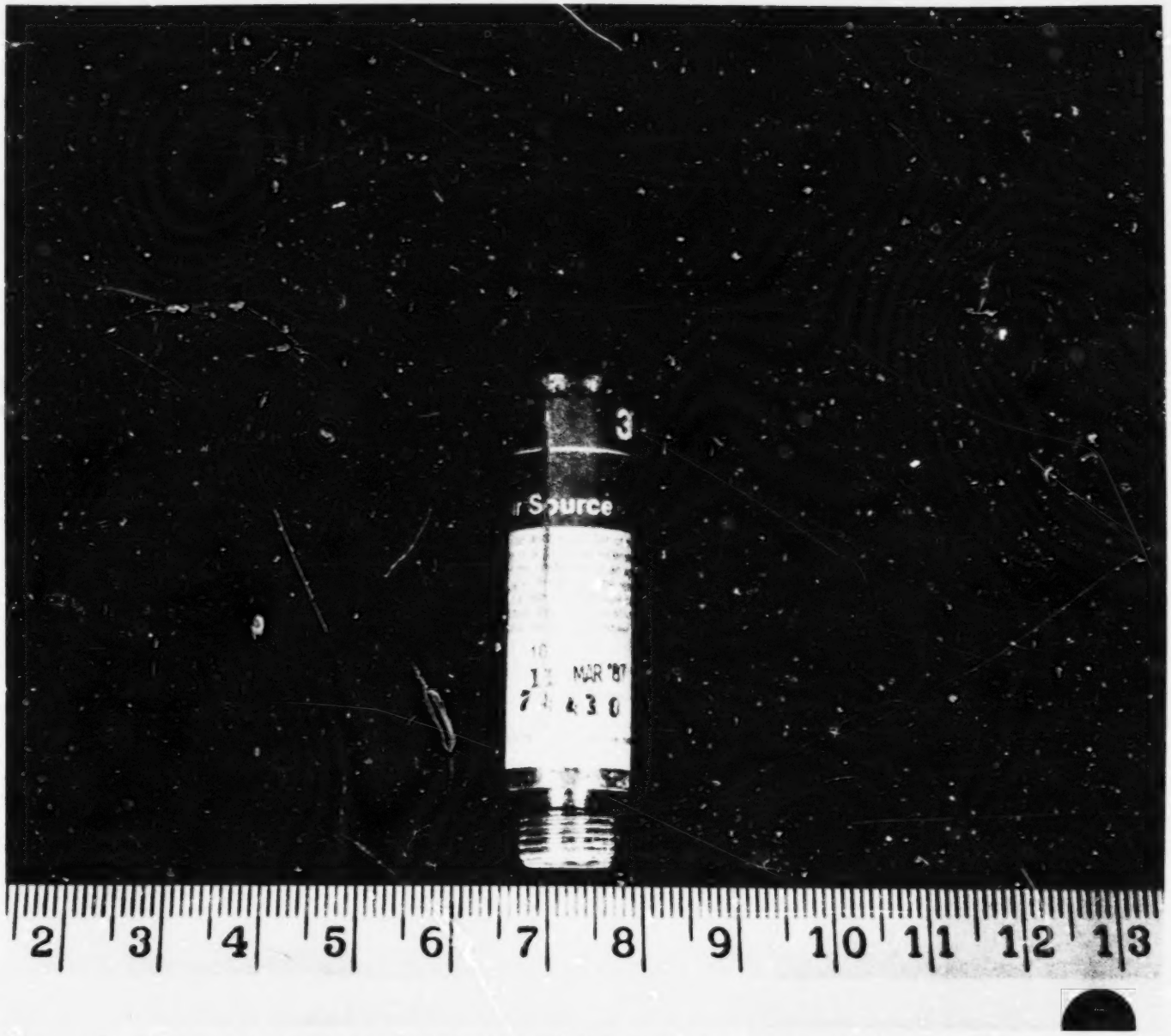


Figure 5 Photograph of static eliminator D74410 from the manual filling station.

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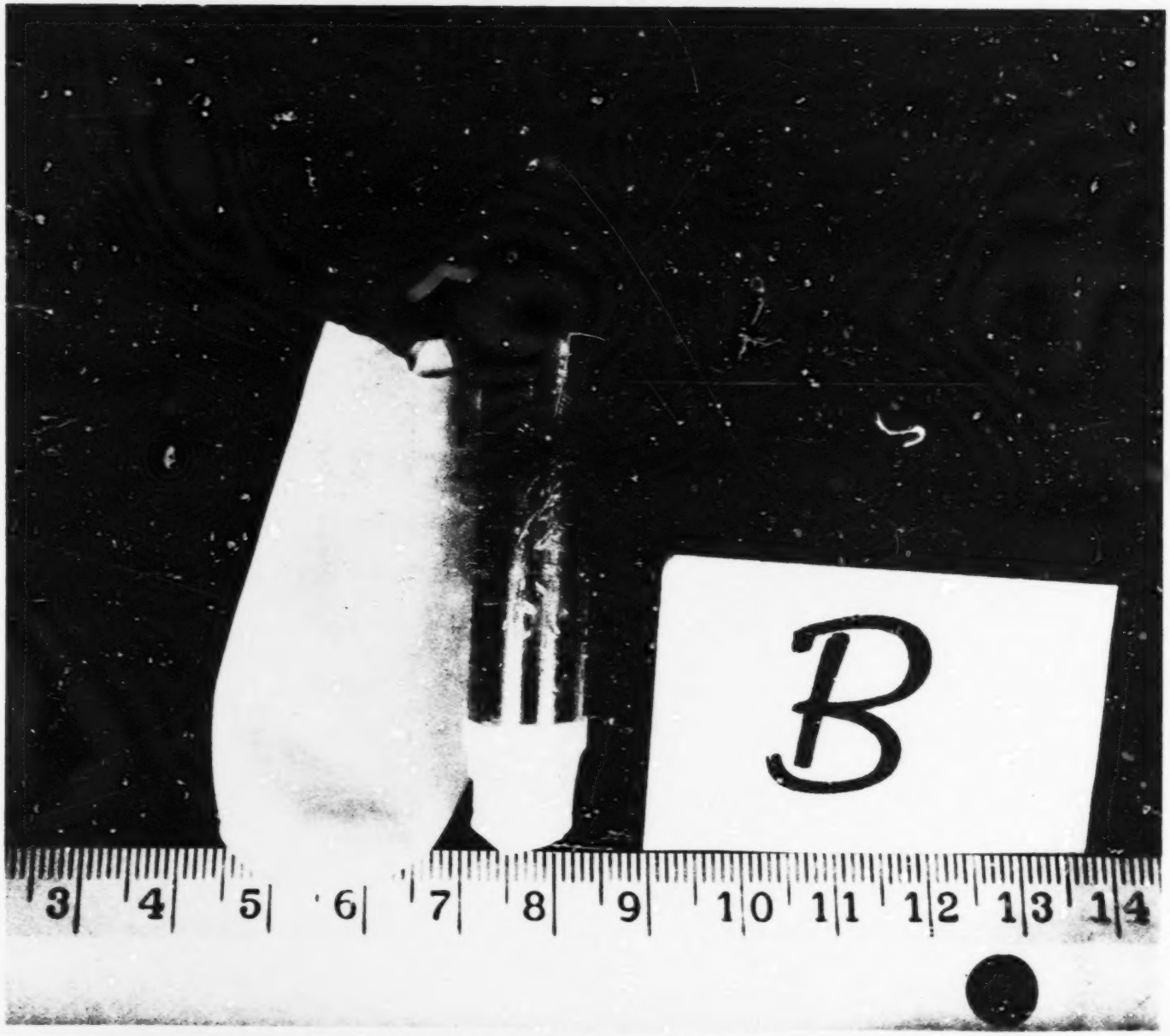


Figure 6 Photograph of device identified as "B" , from the bottle washing facility. Note the scratches/gouges on the casing.

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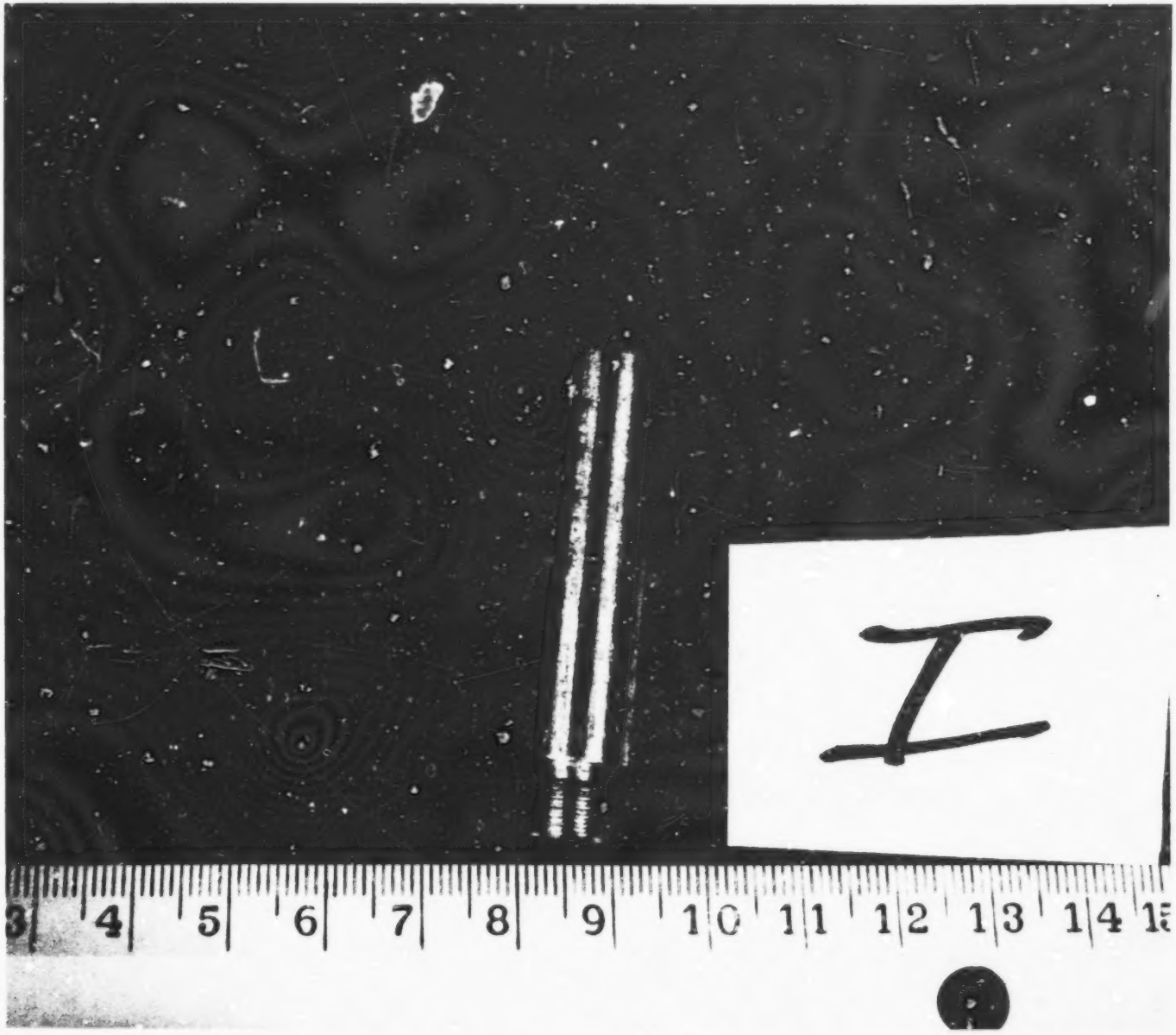


Figure 7 Photograph of static eliminator "I" . Note the scratches on the surface.

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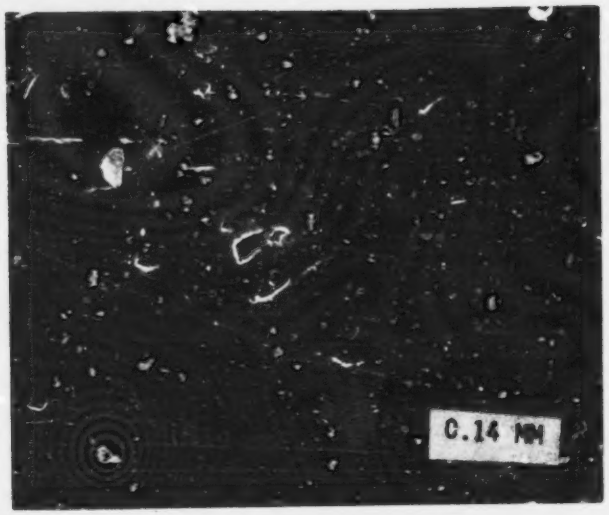
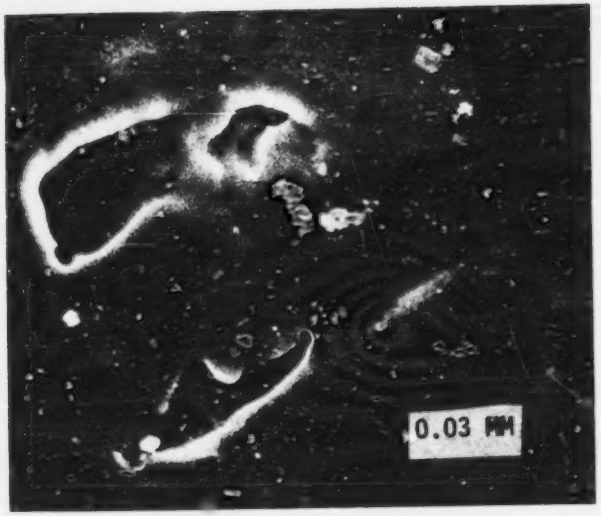



130X

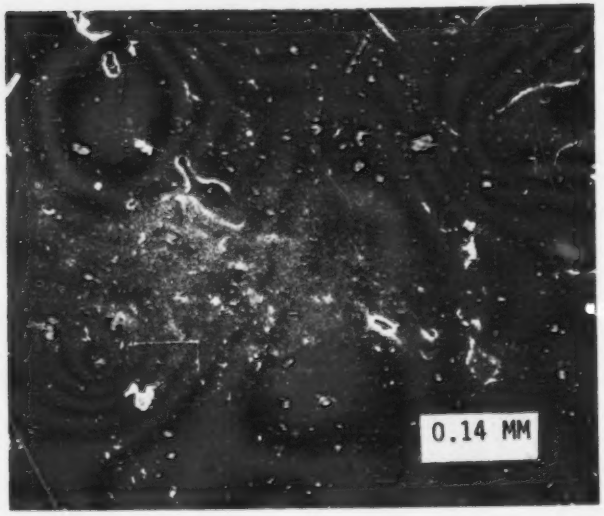
Figure 8 Low magnification SEM photo of the amber epoxy.




900X

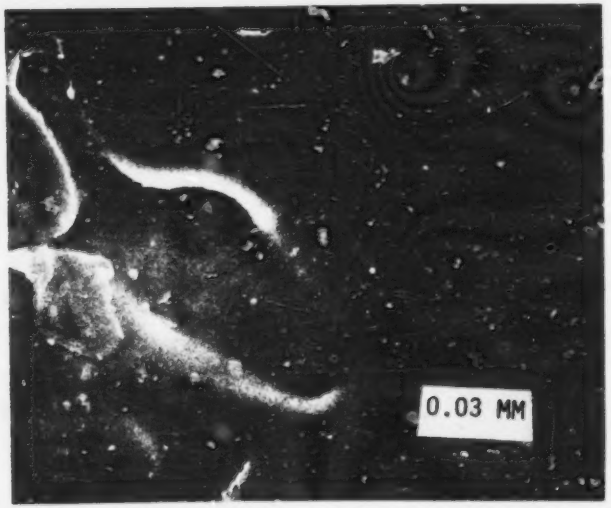
Figure 9 Higher magnification SEM photo showing no areas of cracking in the epoxy.






130X

Figure 10 SEM photo of black epoxy.




900X

Figure 11 No cracks were visible in the black epoxy at higher magnification.

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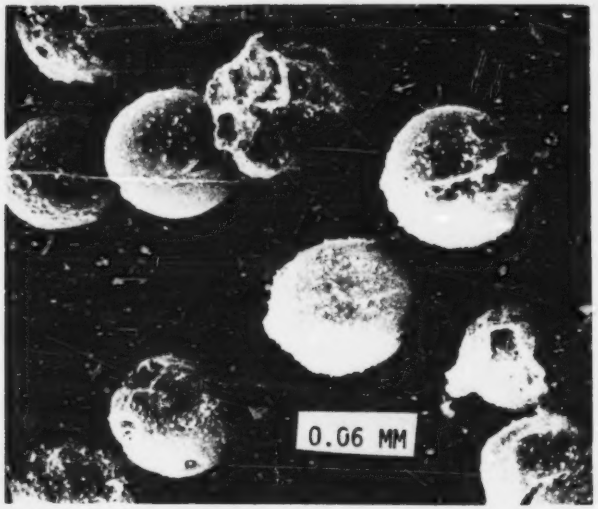
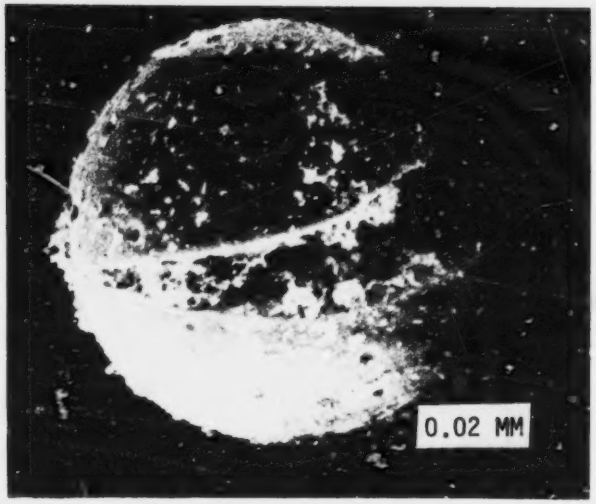



450X

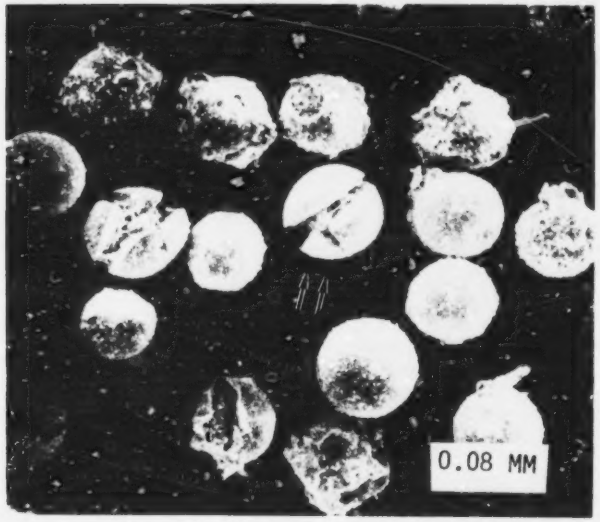
Figure 12 SEM photo of "new" non-radioactive microspheres.




1350X

Figure 13 Higher magnification photo showing cracked microsphere.






315X

Figure 14 A second field also showed cracked microspheres.

1350X

Figure 15 Higher magnification SEM photo depicting cracking of the microsphere.

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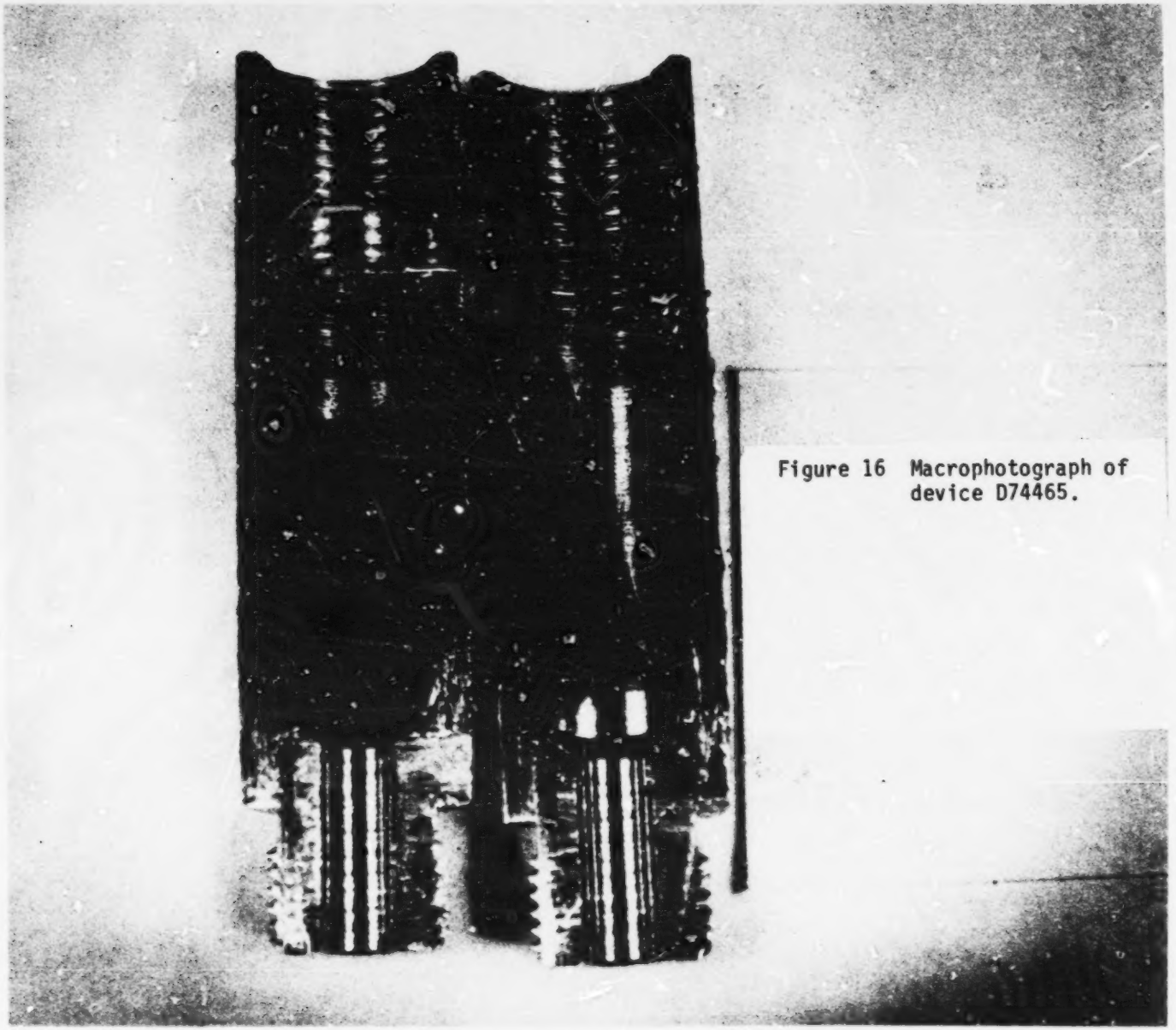

Figure 16 Macrophotograph of device D74465.



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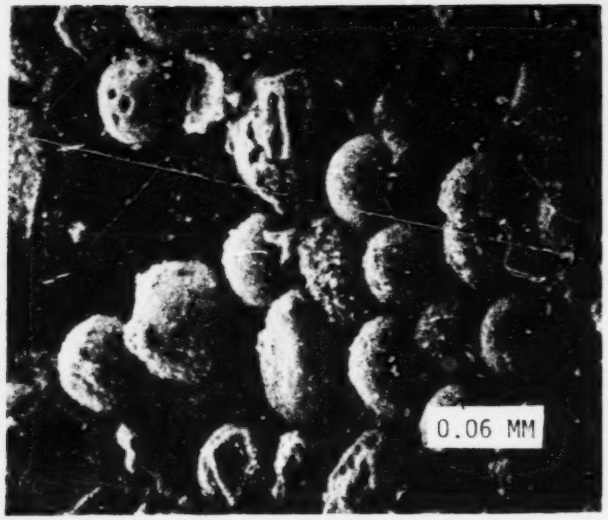
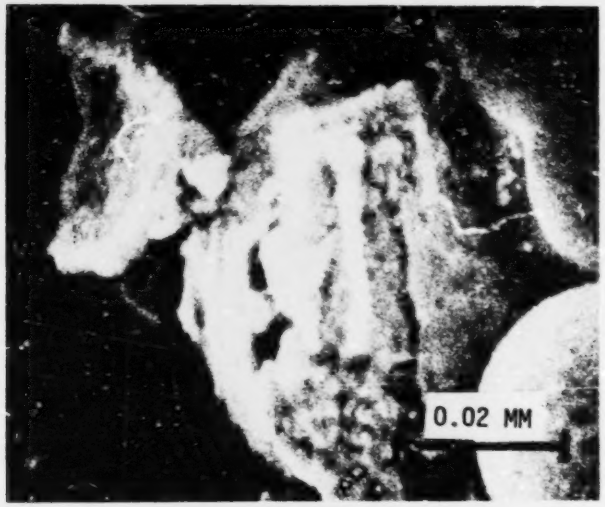



400X

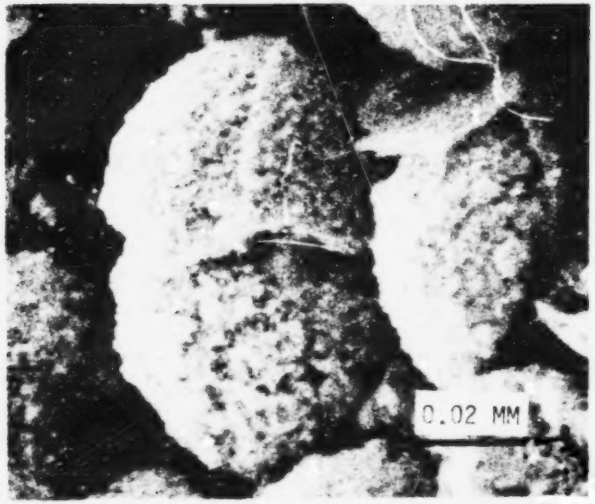
Figure 17 Low magnification SEM photo of a typical area on static eliminator D74465.




1200X

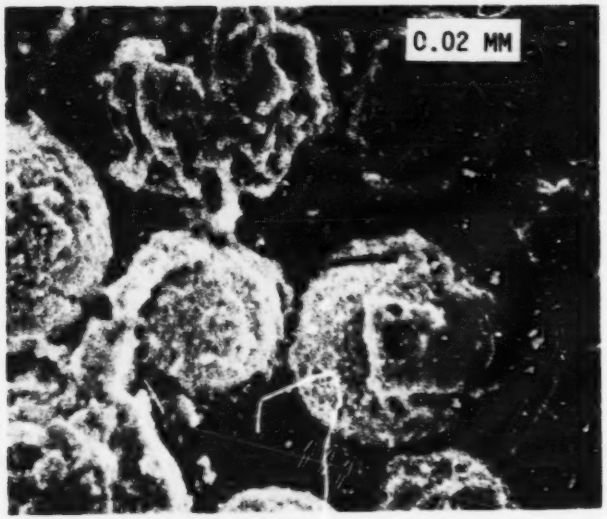
Figure 18 Higher magnification fractograph of a damaged microsphere.






1200X

Figure 19 A second microsphere appeared to be cracked.




1100X

Figure 20 Another area of D74465 showing apparently damaged microspheres and possible decohesion around sphere - arrows.

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Figure 21 Macrophotograph of new device D74471.

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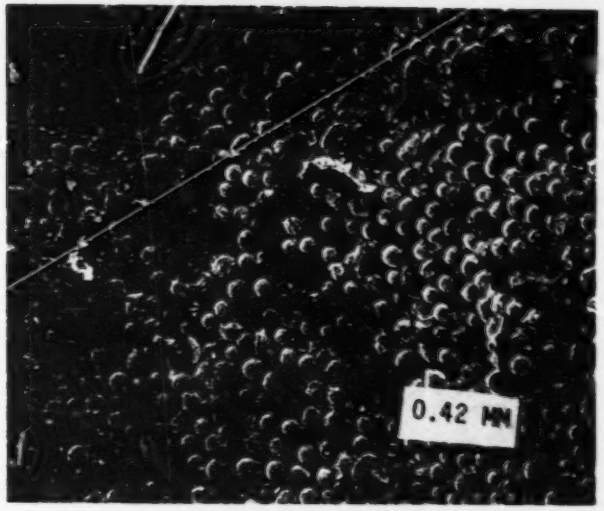
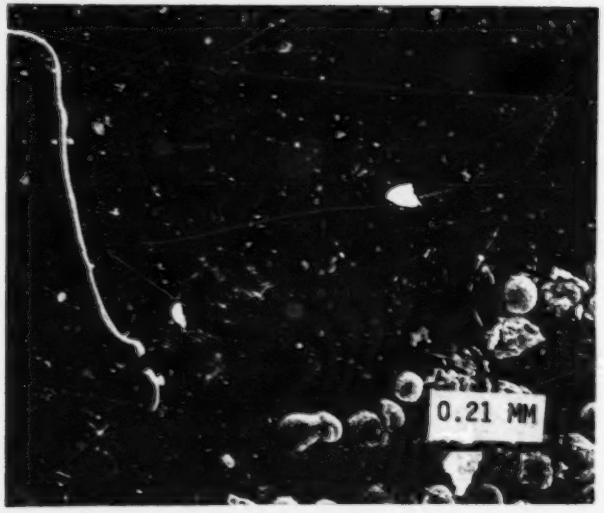



60X

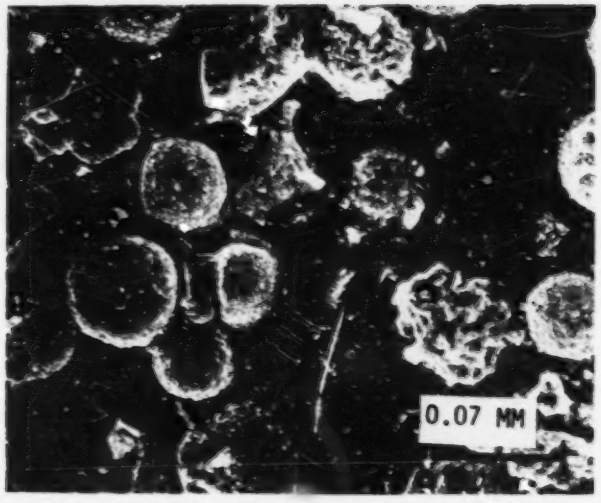
Figure 22 Low magnification fractograph of static eliminator D74471.




120X

Figure 23 Cracking of the epoxy was observed on this static eliminator.






350X

Figure 24 Some decohesion and microsphere damage (arrows) was noted on this specimen.

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Figure 25 The brass jacketed device D64601 is seen in this photograph.



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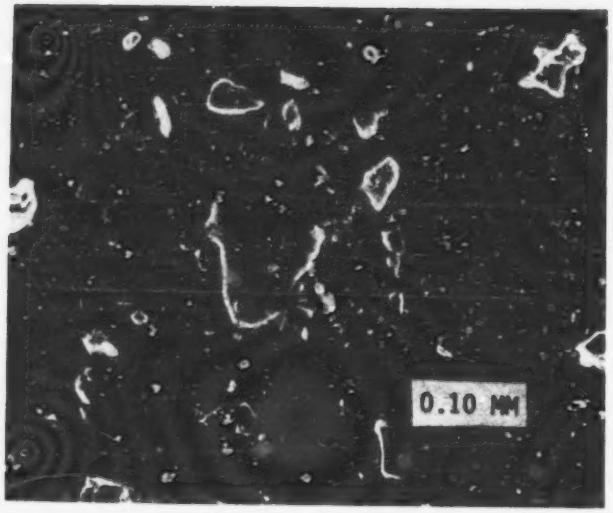
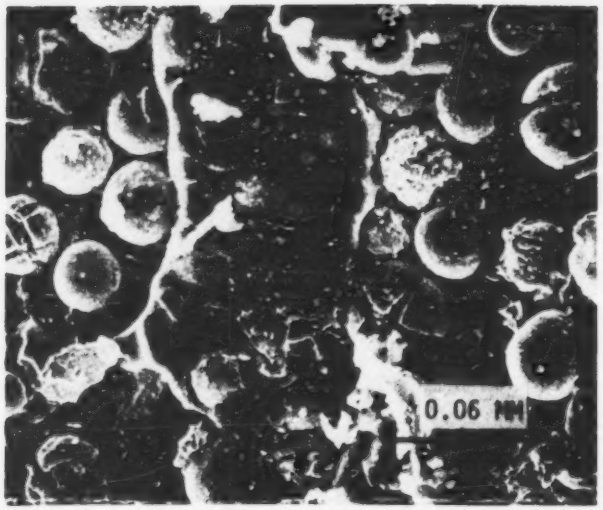



260X

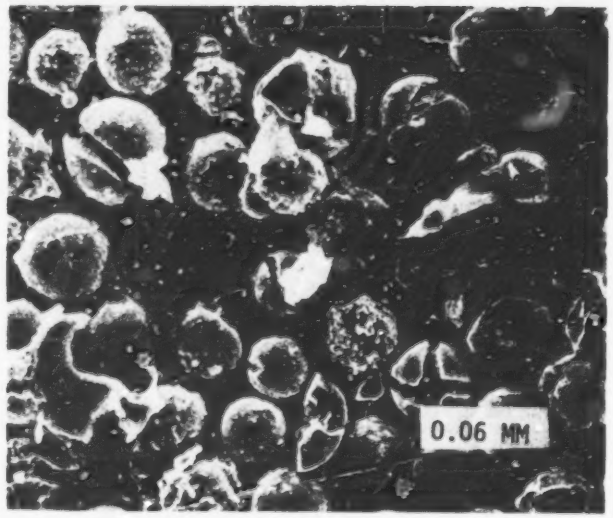
Figure 26 SEM photo of epoxy (typical) on static eliminator D64601.




400X

Figure 27 Fractograph showing decohesion of microspheres (arrows) and damaged microspheres.






400X

Figure 28 A second area showed cracking epoxy in area of the microspheres (D64601).

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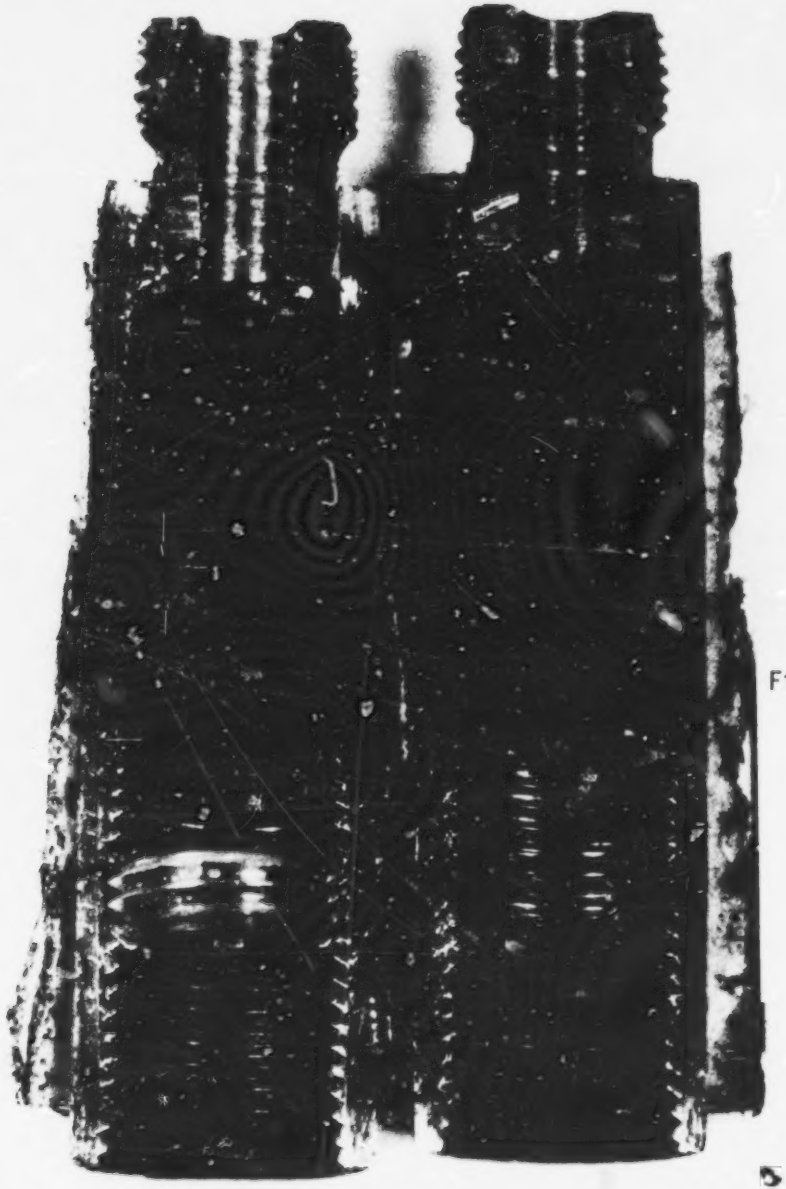

Figure 29 Device D74410 from the manual filling station is seen here. The arrows show an area of missing epoxy.

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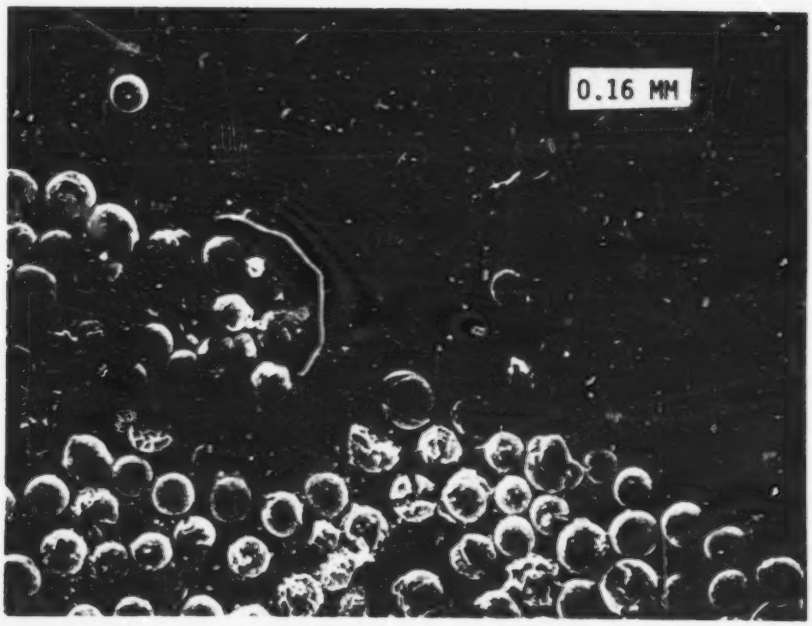
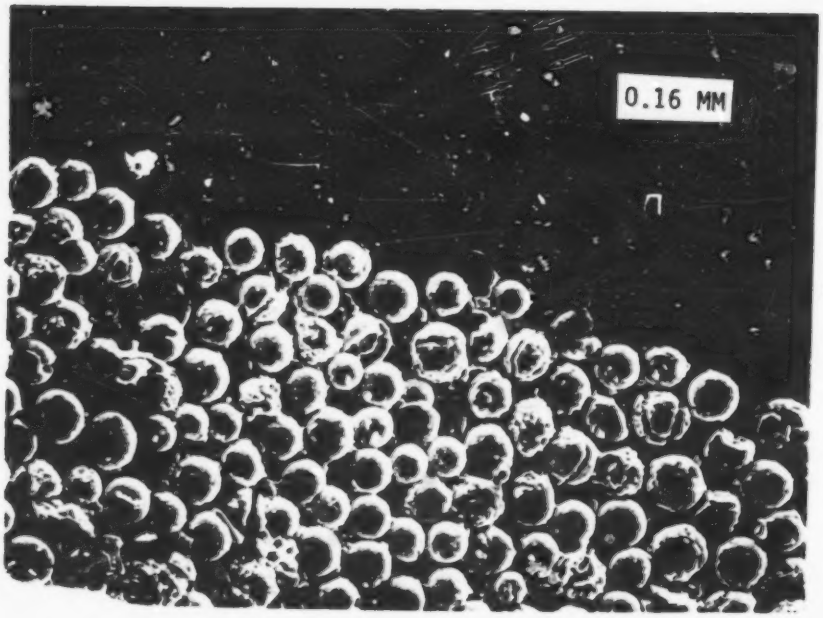



160X

Figure 30 Significant cracking of the epoxy is shown on D74410.




160X

Figure 31 A second area of D74410 showing the typical condition of epoxy and microsphere damage.



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Figure 32 The discoloration of device "B" is evident in this photograph.

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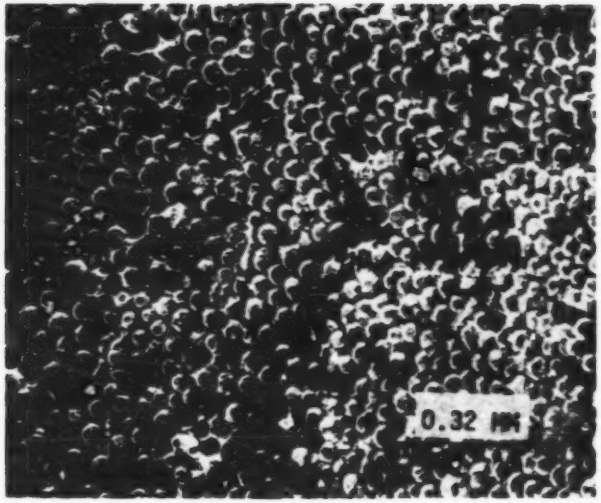
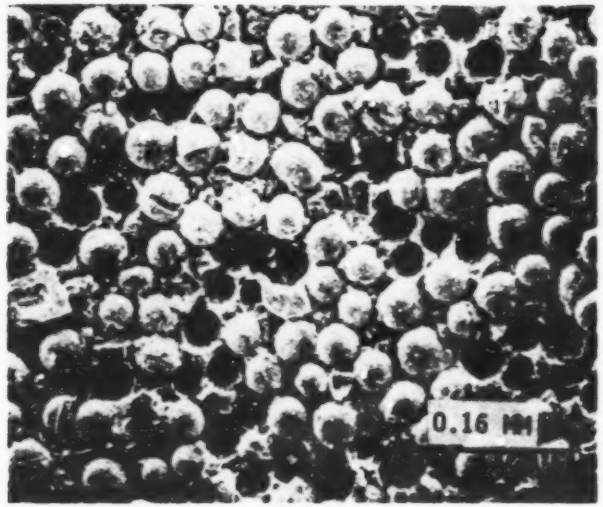


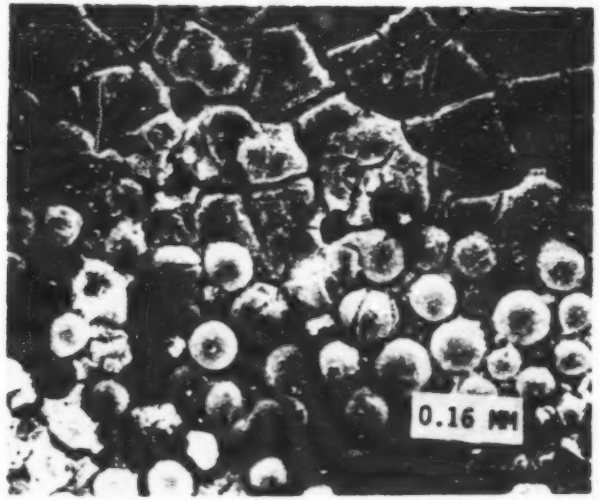

30X

Figure 33 Low magnification SEM photograph showing missing microspheres (dark dots).




160X

Figure 34 This higher magnification fractograph showing the areas of missing microspheres.




160X

Figure 35 Cracked microspheres and a definite "reptile skin" cracking of the epoxy is seen in this fractograph.



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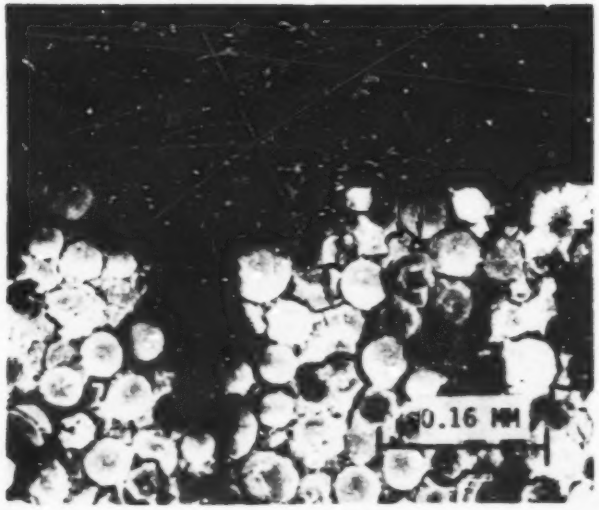
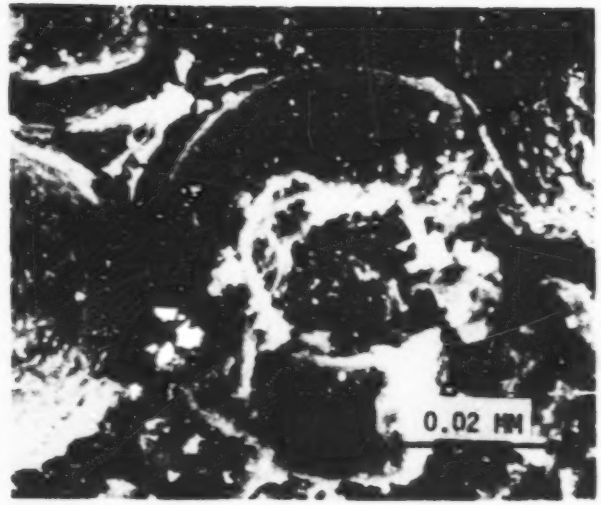


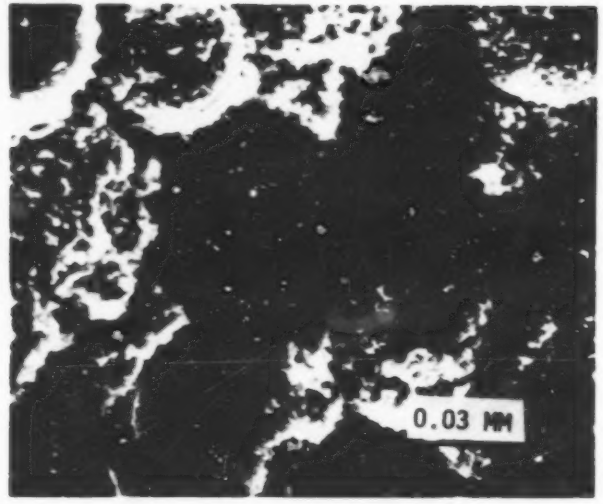

160X

Figure 36 Low magnification SEM photo (sample B) of another area showing damage to both epoxy and microspheres.




1200X

Figure 37 Higher magnification SEM photo of damaged microsphere (Figure 36-arrows).




800X

Figure 38 SEM photo of an area where a microsphere is missing - no apparent swelling of epoxy.



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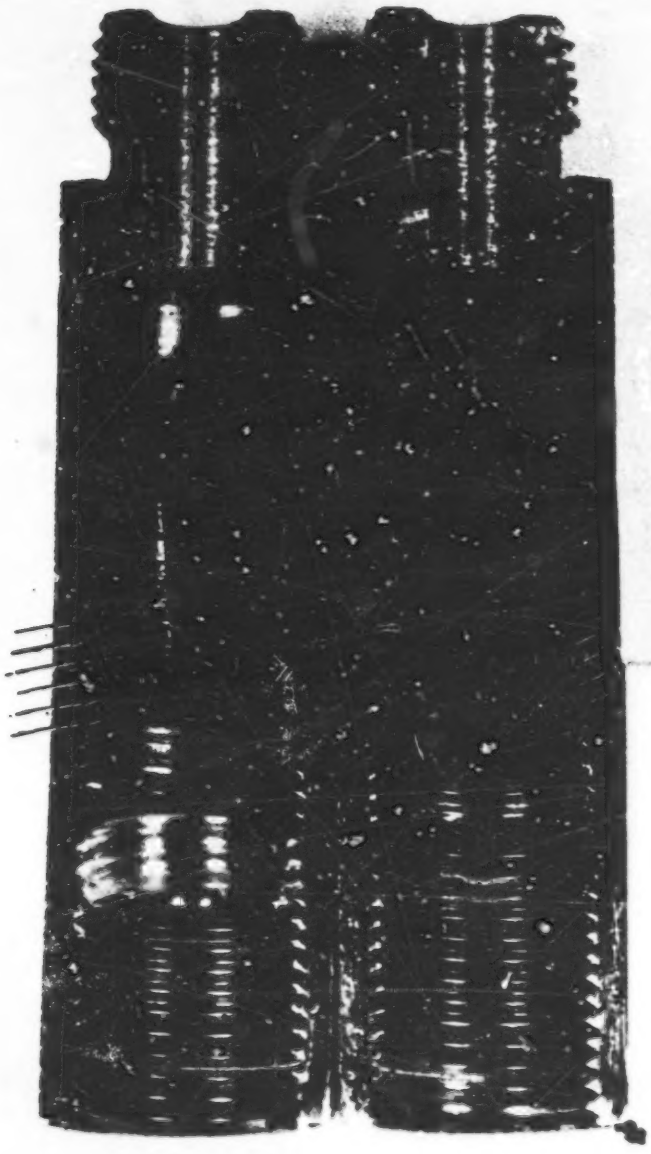

Figure 39 Cracking of the discolored areas of the epoxy are seen here (arrows). Device "I."

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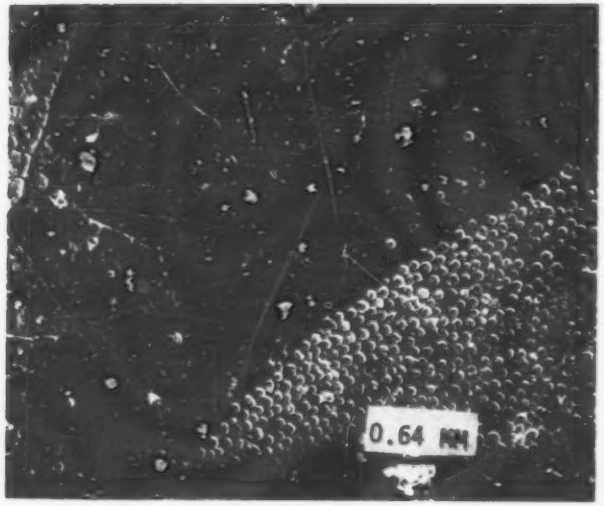
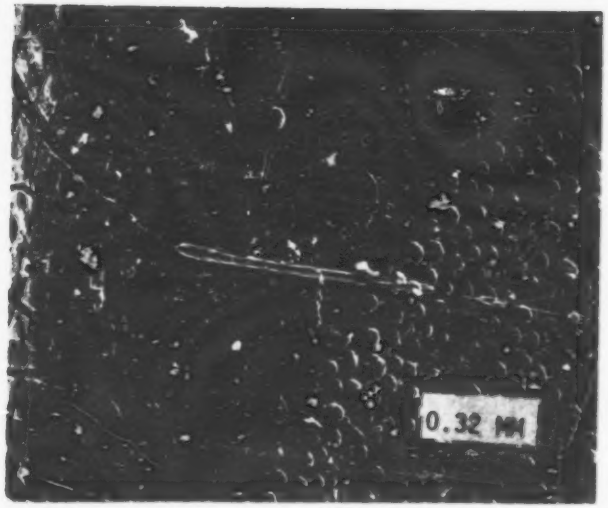


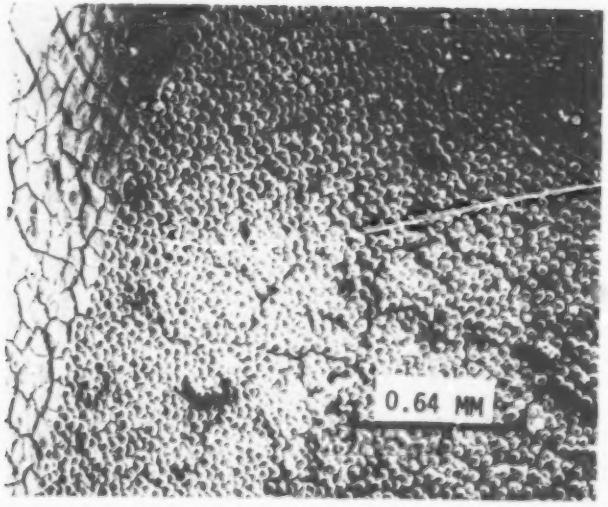

40X

Figure 40 SEM photo of "reptile skin" effect on specimen I.




80X

Figure 41 Microspheres noted outside the area of interest (arrow) and craters visible in epoxy.




40X

Figure 42 This fractograph shows that the epoxy under the microspheres is apparently cracking also.



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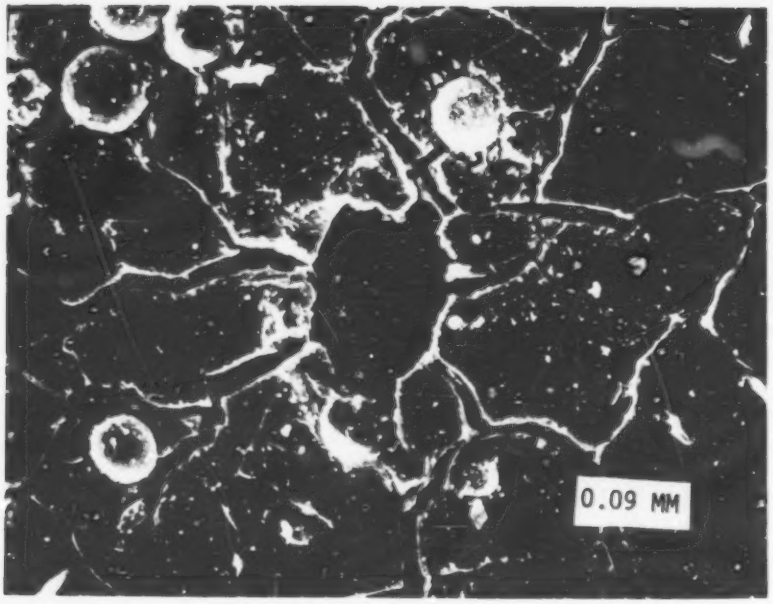
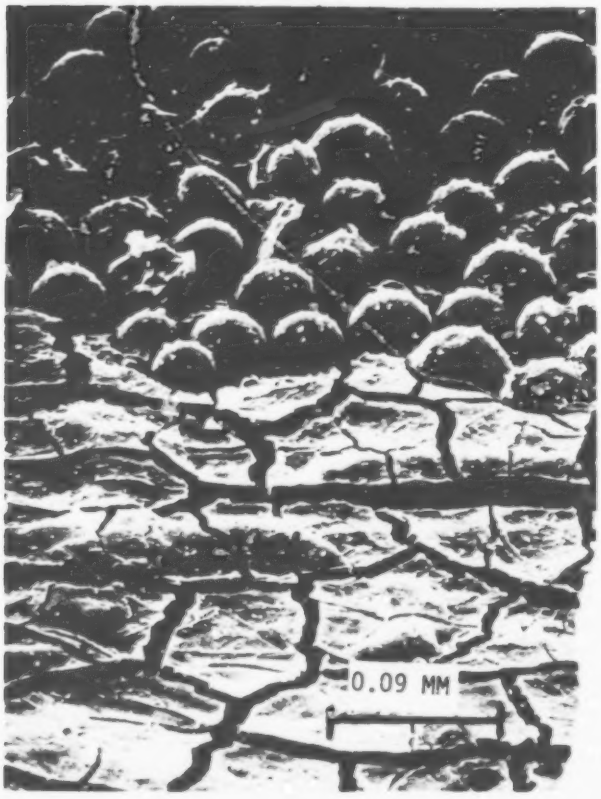



280X

Figure 43 Higher magnification fractograph of a typical crater. Note decohesion around remaining microspheres.




280X

Figure 44 Microsphere damage and epoxy damage are seen in this SEM photo of specimen "I."



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13. ABSTRACT (200 words or less)

Numerous instances of facility contamination by polonium-210 microspheres have been reported. These contamination events appear to be caused by leakage or ejection of the radioactive material from static elimination devices manufactured by the Minnesota Mining and Manufacturing Company (3M). The Ashland Chemical Company (Easton, Pennsylvania) was the first facility determined to be contaminated and was subsequently subjected to a comprehensive review by the U. S. Nuclear Regulatory Commission (USNRC). Therefore, devices from this facility were chosen for examination.

A failure investigation has been performed on six static eliminators. The investigation consisted of visual inspection, sectioning and scanning electron microscopy of six devices. It is concluded that non-uniform and imperfect microspheres appear to have been manufactured and installed in devices. Handling incident to use may induce loosening of the microspheres and the epoxy binder used in device manufacture may not be suitable for the varied service conditions.

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